IN THE UNITED STATES DISTRICT COURT
FOR THE NORTHERN DISTRICT OF ILLINOIS
EASTERN DIVISION

DOCKETED
AUG 2 0 2002

PALL CORPORATION

Plaintiff,

v.

ENVIRONMENTAL AND POWER TECHNOLOGIES, LTD. and PETER DUFRESNE

Defendants.

Civil Action No. 02C 5898

JUDGE GOTTSCHALL

MAGISTRATE JUDGE SCHENKIER

COMPLAINT

Plaintiff Pall Corporation ("Pall"), by its undersigned attorneys, for its claims for relief against Defendants Environmental and Power Technologies, Inc. ("EPT") and Peter Dufresne ("Dufresne"), alleges as follows:

JURISDICTION AND VENUE

1. This is an action for a declaratory judgment that Reexamined United States Patent B1 5,661,117 ("the '117 patent") is invalid, unenforceable and not infringed by any process or product made, used, offered for sale, imported or sold by Plaintiff Pall, or by customers or distributors of Pall's Ion Exchange Systems or by any end user of Pall's Ion Exchange Systems manufactured by Pall (hereinafter collectively referred to as "Pall customers").

2. This action is also brought for damages and injunctive relief as a result of the Defendants' patent misuse, trade disparagement, tortious interference with contractual

relations and prospective business advantage, violations of the Lanham Act (15 U.S.C. Sections 1051 et seq.) and unfair competition.

3. This Court has jurisdiction over the claims of the declaratory judgment action arising under the patent laws of the United States, 35 U.S.C. Sections 1 et seq. (Counts I and II) pursuant to 28 U.S.C. Section 1338 and 28 U.S.C. Sections 2201 and 2202, and for the unfair competition claim (Count VI) pursuant to 28 U.S.C. Section 1338(b). This Court has jurisdiction over the claims for relief arising under the Lanham Act 15 U.S.C. Sections 1051 et seq. (Count IV) pursuant to 15 U.S.C. Section 1121. This Court further has supplemental jurisdiction over the remaining claims for relief under state law and common law (Counts III and V) pursuant to 28 U.S.C. Section 1367.

4. Venue is proper in this district pursuant to 28 U.S.C. Section 1391 because the Defendants, and each of them, are transacting and doing business and conducting or otherwise transacting its affairs in this district and/or a substantial part of the events giving rise to the claims in this action occurred in this district.

5. EPT and Dufresne have specifically and purposefully directed their activities to Illinois generally, and this judicial district specifically, by committing certain of the tortious acts, described below, in this judicial district.

PARTIES

6. Pall is a corporation organized and existing under the laws of the State of New York, with its principal office in East Hills, New York.

7. EPT, upon information and belief, is a corporation organized under the laws of Canada, with its principal place of business at 4712-13th Street N.E., #109, Calgary, Alberta, Canada, T2E 6P1.

8. Dufresne, upon information and belief, is a citizen of Canada. Dufresne's business address is 4712-13th Street N.E., #106, Calgary, Alberta, Canada, T2E 6P1. On information and belief, Dufresne is the Owner, President and General Manager of Company Facilities at EPT.

GENERAL ALLEGATIONS

Background Of The Patent-In-Suit

9. Dufresne, by and through a patent agent, filed an original patent application, entitled "Regeneration Of Phosphate Ester Lubricating Fluids," with the United States Patent and Trademark Office ("PTO") on April 14, 1995. The PTO assigned Patent Application Serial Number 08/421,771 to this application ("the '771 application").

10. Along with the '771 application, Dufresne further submitted to the PTO an Information Disclosure Statement ("the original IDS") that, according to its terms, was intended to "comply with the duty of disclosure requirements of 37 C.F.R. 1.56." The original IDS disclosed two patents: U.S. Patent No. 4,741,857, which was issued to Horwitz et al. (the "Horwitz prior art"), and U.S. Patent No. 3,708,508, which was issued to Schulz (the "Schulz prior art").

11. Upon initial consideration of the '771 application, the PTO rejected all patent claims as anticipated, under 35 U.S.C. §102(b), or as obvious, under 35 U.S.C. §103, in view of the Horwitz prior art and the Schulz prior art.

12. On July 11, 1996, Dufresne responded to this initial examination by submitting an Amendment ("the First Amendment"), which had the effect of amending the original claims and adding new claims. The amended and new claims included limitations

that were not in the original claims. The First Amendment also canceled certain claims from the '771 application.

13. In the remarks section of the First Amendment, Dufresne argued that his purported invention could be distinguished from the Horwitz and Schulz prior art primarily because the amended patent claims required the contact of phosphate ester lubricant fluid with an anionic resin and no "further treatment" of the material. The First Amendment specifically included the following statement to the PTO: "With Applicant's arrangement, the process involves the contact of a phosphate ester lubricant fluid with an anionic resin. No further treatment of the material is required."

14. After considering the First Amendment, the PTO again rejected all of the amended claims and Dufresne's arguments as not persuasive. The PTO specifically recognized that the Horwitz prior art described contacts between an extractant and an exchange system that consisted of both cationic and anionic resins. The PTO also concluded that Dufresne's claims, even as amended, had not excluded the possibility of treatment after the extractant had contacted the anionic resin.

15. Dufresne responded to the second rejection of the '771 application by again amending his patent claims. In an Amendment filed with the PTO on January 20, 1997 ("the Second Amendment"), Dufresne substituted the phrase "said method consisting essentially of" for the phrase "said method comprising" in the existing claims. The Second Amendment, by way of the new phraseology, limited the scope of the claimed subject matter to only the specific limitations recited in the claim.

16. In the remarks section of the Second Amendment, Dufresne also argued that the second set of amendments limited the claims to passing phosphate ester lubricant through only an "anionic resin:"

> Further, claim 20 is now limited to the step of passing phosphate ester lubricant through a source of anionic resin. This closure in the scope of the claim has been indicated by the use of the language "consisting essentially of".
>
> ***
>
> Regarding claim 21 similar restrictions are incorporated in this claim. This claim is obviously restricted to a polystyrene anionic resin, a specific group of metal contaminants, acid contaminants, and similar to claim 20 is a closed method claim.

17. Dufresne also argued that his amended claims were patentably distinct from the Horwitz prior art because the Horwitz reference "is limited to a combination of a cationic and anionic treatment regime."

18. In response to the limiting claim amendments and arguments made by Dufresne, the PTO issued a Notice of Allowability as to the amended claims on February 18, 1997. According to the Notice of Allowability, the issue fee payable to the Patent Office for issuance of the patent was due on May 19, 1997.

19. Based on his claim amendments and arguments to the PTO, Dufresne surrendered from the scope of the patent-in-suit any claim to an ion exchange system that uses a combination of anionic and cationic resins to filter contaminants.

20. On or about February 20, 1997, and after the PTO had issued the Notice of Allowability, Dufresne submitted a second Information Disclosure Statement to the PTO ("the Second IDS"). The Second IDS identified a Patent Cooperation Treaty (PCT) application, PCT/GB94/00954 entitled "Fluid Treatment Process" by William Phillips ("the Phillips PCT application"), as a prior art reference against Dufresne's '771 application.

21. The Second IDS stated that "although there are general similarities to Applicant's invention, it is clear that the publication does not disclose the important features of the total acid number, new fluid quality, or the removal of contaminants selected from the group consisting of aluminum, chromium, tin, iron, sodium, calcium, magnesium, silicon and phosphorous based contaminants."

22. Contrary to Dufresne's assertions in the Second IDS, the Phillips PCT application, at a minimum, discloses the total acid number and removal of contaminants from the group consisting of aluminum, iron, sodium, calcium, magnesium and copper.

23. The Second IDS further stated that "[t]he reference submitted does not anticipate nor render obvious the steps in the claimed process and therefor is submitted herewith for the purpose of candor."

24. Dufresne, by and through his patent agent, further submitted a "Certification Under 37 C.F.R. § 1.97(e)" that purported to explain the reason why the Phillips PCT application had been cited at that particular point in time. Dufresne's patent agent stated that Dufresne had become aware of the Phillips PCT application "approximately one month" prior to the submission of the Second IDS. This certification further stated "Applicant's representative only received this reference February 10, 1997, and has therefor attempted to advance the prosecution of the case by immediately supplying the Examiner with this reference."

25. Based on the facts stated in the "Certification," Dufresne had knowledge of the Phillips PCT application prior to the date the PTO issued the Notice of Allowability in the '771 application, but he did not submit this reference for consideration until after this event.

26. The PTO rules in effect at the time that Dufresne submitted the Second IDS required the following from all applicants that sought to bring prior art to the attention of the PTO after the Notice of Allowance issued but prior to payment of the issue fee: (1) the certification required by 37 C.F.R. § 1.97(e), (2) a petition requesting consideration of the information disclosure statement, and (3) payment of an appropriate fee.

27. The Second IDS did not include the papers and fees required by the PTO rules. As such, the Second IDS was defective and did not disclose the Phillips PCT application to the PTO.

28. The PTO rules in effect at the time that Dufresne submitted the Second IDS precluded an Examiner from considering prior art submitted in an information disclosure statement after payment of an issue fee.

29. On or about May 9, 1997, Dufresne paid the issue fee in the '771 application.

30. By submitting a defective information disclosure statement and by paying the issue fee without correcting the defective information disclosure statement, Dufresne ensured that the PTO would not consider the Phillips PCT application during the prosecution of the '771 application.

31. On or about July 8, 1997, the PTO informed Dufresne that the Second IDS would not be considered in connection with the '771 application because Dufresne had not submitted the necessary petition, fee, and a certification of mailing. The PTO further informed Dufresne that he could withdraw the '771 application from issuance in order to permit the PTO to consider the Phillips PCT application.

32. Dufresne did not withdraw his '771 application from issuance, and thereby denied the PTO an opportunity to consider the Phillips PCT application in connection with the claims presented in the '771 application.

33. On August 26, 1997, the PTO issued the '117 patent. It did not consider the effect of the Phillips PCT application on the allowed and issued claims in that patent. The face of the '117 patent identifies Dufresne as the sole inventor of the invention disclosed therein. A copy of the '117 patent is attached hereto as Exhibit A.

34. On August 26, 1998, exactly one year to the day after the PTO issued the '117 patent, a third-party request for reexamination of the '117 patent was filed with the PTO. The reexamination request was based on, among other prior art, the Phillips PCT application.

35. The PTO granted the third-party's request for reexamination on or about October 29, 1998, and assigned Reexamination Control No. 90/005,088 to the new application ("the '088 application"). The PTO relied on the Phillips PCT application, among other prior art, as a reason for granting the reexamination request.

36. On or about March 4, 1999, the PTO rejected eight of the original sixteen claims "as being clearly anticipated by Phillips (PCT Publication WO 94/2550)."

37. Dufresne subsequently submitted an Amendment, on or about July 27, 1999 ("the Third Amendment"), that conceded the PTO's rejection of its claims on the basis of the Phillips PCT application. By way of the Third Amendment, Dufresne specifically canceled four claims and amended the remainder of the claims to include specific numerical limitations.

38. Dufresne's cancellation of previously allowed claims in response to the PTO's rejection is an admission that the Phillips PCT application was material prior art that Dufresne should have submitted to the PTO in connection with the '771 application.

39. Dufresne's Third Amendment further limited all claims to a process of cleaning a phosphate ester lubricant fluid with a replaceable filter containing an anionic resin and wherein the "moisture content" of the anionic resin was "of at least about 50%." In addition, Dufresne further limited all claims to require that the replaceable filter be replaced "at a periodicity of up to 27 months" and when there is an increase in the "total acid number" ("TAN") of the fluid above 0.07.

40. The Third Amendment also argued that the numerical limitations specified by the claim amendments produced "new and unexpected results." Dufresne specifically argued that the "criticality of the newly added limitations" supported the patentability of the claims because "[t]he crux of the patentable subject matter of U.S. Patent No. 5,661,117 inheres in the unexpected and surprising results achieved when a particular resin filter is both provided to and systematically precisely replaced in connection with a phosphate ester lubricant system."

41. In a further written submission to the PTO, Dufresne supplemented the Third Amendment with further argument and a declaration that he filed on September 7, 1999. In this supplemental submission, Dufresne again argued that the newly added numerical limits supported a finding that the amended claims were patentable over the Phillips PCT application. He specifically stated that "[t]he critical aspect of the inventive method inheres not merely in the use of an anionic resin filter to maintain phosphate ester lubricant, but

specifically resides in observance of the parameters and conditions within which the resin filter must be replaced."

42. Dufresne's supplemental submission further included data and "evidence" to support his argument regarding the numerical limitations. According to Dufresne, "[a]ll of the data support the necessity of changing the anionic resin filter when TAN exceeds 0.07 and replacing it with a filter having at least about 50% moisture." He further concluded that "data identifying TAN plotted against time illustrate that failure timely to change the resin when TAN exceeds 0.07 results in drastic, rapid increases in TAN levels (and concomitant lubricant degradation)."

43. The September 7, 1999, supplemental submission included an "Expert's Declaration of Peter Dufresne," which further included attached spreadsheets containing numerical data. Dufresne's declaration refers to an "'alarm action'" and further states that "the spreadsheet evidences the importance of maintaining TAN below about 0.07 and accomplishing doing so with anionic resin cartridges having a minimum moisture content of 50%."

44. On or about May 18, 2000, the PTO issued a Notice of Intent to Issue Reexamination Certificate. In the accompanying Statement of Reasons for Patentability and/or Confirmation, the PTO Examiner stated that "the Expert Declaration by applicant shows the unexpected results in phosphate lubricant maintenance when the resin has the claimed moisture content and the criticality of the claimed conditions for replacement of the resin filter."

45. Based on his claim amendments and arguments to the PTO in the Third Amendment, Dufresne surrendered from the scope of the patent-in-suit any claim to use an

ion exchange system in which a replaceable filter is not replaced precisely within the parameters specified by the claims.

46. On August 29, 2000, the PTO issued Reexamination Certificate B1 5,661,117 ("the Reexamination Certificate"). Unless otherwise specified, the '117 patent and the Reexamination Certificate are hereinafter collectively referred to as "the '117 patent." A copy of the Reexamination Certificate is attached hereto as Exhibit B.

Defendants' Threats And Charges Against Pall And Pall's Customers

47. On April 4, 2002, EPT sent a letter to Great Lakes Chemicals ("GLC"). The April 4, 2002, letter was signed by Dufresne, in his capacity as General Manager of EPT, and addressed to David Phillips, an employee of GLC. In the April 4, 2002, letter, Dufresne stated the following:

> We have had some interesting developments in regards to the PALL Corp selling IX equipment. We have taken the approach of going after their customers instead of PALL. Their customers have a "zero-tolerance" to legal liability especially after our Patent was appealed and upheld. You will shortly see a whosale [sic] return these PALL units back to PALL.

48. The April 4, 2002, letter further referred to an offer that EPT had purportedly extended to Akzo Nobel to purchase the patent-in-suit for $5,000,000. Dufresne offered to sell to GLC the patent-in-suit for the same price. The April 4, 2002, letter suggested that GLC might have some "financial interest" in the offer because "we will establish a stranglehold on the PALL Corporation."

49. In a series of letters in early 2002, EPT made good on its threat of "going after" Pall's customers. The series of letters, signed by Dufresne in his capacity as President of EPT, specifically threatened several of Pall's customers that their use of Pall's Ion Exchange Systems would infringe the '117 patent.

50. In a letter dated April 24, 2002, to John Abdelmalak ("Abdelmalak") of the Los Angeles Department of Water and Power, Dufresne made the following false statement regarding the patent-in-suit: "The patent gives my company the exclusive right to sell, market, use and manufacture ion exchange conditioning systems for use on phosphate ester fluid." The April 24, 2002, letter to Abdelmalak further falsely stated: "Using a Pall ion exchange system would be in direct infringement of our patent rights as this equipment would be using ion exchange resins, specifically (anion resins) which the patent includes." Dufresne's letter concluded with the suggestion that returning Pall's products would be "the easiest way to discharge your infringement liability."

51. Early in 2002, EPT sent a letter to Exelon Corporation, which is located in this judicial district. On information and belief, the Exelon letter was substantially similar to the April 24, 2002, letter sent to the Los Angeles Department of Water and Power. (See ¶ 50).

52. EPT and Dufresne have caused communications to be made to Pall and Pall customers, asserting, without adequate basis in fact or law, and without making a reasonable investigation, generally that Pall's Ion Exchange Systems, and processes utilizing such systems, infringe the '117 patent.

53. EPT and Dufresne, in bad faith and without reasonable investigation, have knowingly and recklessly asserted infringement based on the use of Pall's Ion Exchange Systems. These statements were made to Pall's customers to the detriment of Pall and competition, have caused Pall's customers to express concern to Pall regarding the infringement assertions, and have otherwise disrupted the business of Pall.

54. On May 17, 2002, Pall advised EPT and Dufresne in a letter that it had studied the matter and concluded that Pall's Ion Exchange Systems do not infringe the '117 patent.

Pall requested that EPT and Dufresne stop sending threatening letters to end users of Pall's Ion Exchange Systems.

55. On May 17, 2002, EPT and Dufresne responded by again asserting, in bad faith, that "Pall Corporation is in violation of our United States Patent." The May 17, 2002, letter concluded with the statement that "we will take corrective action as required."

56. On information and belief, EPT and Dufresne do not hold a good faith belief that Pall and/or Pall's customers are infringing a valid and enforceable patent.

57. While EPT and Dufresne claim they have investigated whether Pall infringes the '117 patent, they have failed to conduct a reasonable investigation or have failed to conduct any investigation at all, or if they have conducted an investigation, then they know or should know that Pall does not infringe the '117 patent. Defendants have no reasonable basis for concluding otherwise.

58. Pall is not infringing any valid, enforceable patent or patent claim of EPT or Dufresne.

59. EPT and Dufresne have represented that the '117 patent covers all or virtually all of the technology or products produced for use in phosphate ester fluid ion exchange conditioning systems.

60. EPT and Dufresne contend that no company in the market for phosphate ester fluid ion exchange conditioning systems may continue its business or legitimately remain in the market without a license from EPT and Dufresne. Thus, companies in the relevant market must either pay royalties to EPT and Dufresne thereby increasing the cost of the product or face Defendants' threats of expensive litigation and threats of exclusion from the market.

COUNT I - DECLARATORY JUDGMENT OF NONINFRINGEMENT, INVALIDITY AND UNENFORCEABILITY

61. Pall realleges and incorporates herein by this reference paragraphs 1-60 of this Complaint, as though fully set forth herein.

62. As a result of the correspondence with Pall and Pall's customers, there exists an actual and present justiciable controversy between Pall and Defendants as to:

a. The infringement of any properly interpreted claim of the '117 patent;

b. The right of Defendants to threaten or maintain suit for infringement of the '117 patent against Pall and/or Pall's customers;

c. The validity and scope of the '117 patent;

d. The enforceability of the '117 patent against Pall and/or Pall's customers; and

e. The existence of defenses in favor of Pall and Pall's customers to Defendants' assertions of infringement.

63. The '117 patent is limited by its express terms, by the prior art and by representations made to the PTO by Dufresne during the prosecution of the underlying patent application to first induce the PTO to issue the patent and then to issue the reexamination certificate such that neither Pall nor any Pall customer infringes any properly interpreted claim of the '117 patent.

64. The claims of the '117 patent are invalid and void for failure to meet one or more of the requirements and/or conditions of patentability of one or more of Sections 101, 102, 103 and 112 of Title 35, United States Code.

65. The '117 patent is unenforceable because it was obtained through fraud and/or inequitable conduct on the PTO, and through, inter alia, Dufresne's actions, representations and omissions which include those as described below:

a. intentional failure to timely file with the PTO an information disclosure statement identifying the Phillips PCT application;

b. intentional failure to state the materiality of the Phillips PCT application in the information disclosure statement of February 18, 1997;

c. intentional failure to include a petition requesting consideration of the Phillips PCT application and petition fee along with the information disclosure statement of February 18, 1997;

d. intentional failure to withdraw the '117 patent from issue in order to file a File Wrapper Continuation application; and

e. intentional failure to file with the PTO an unequivocal statement identifying the claims in the '117 patent unpatentable over the Phillips PCT application.

66. EPT and Dufresne knew or should have known that the '117 patent does not apply to Pall's Ion Exchange Systems, and EPT and Dufresne lack a reasonable basis for assertion of infringement by Pall or Pall's customers. The enforcement of the '117 patent against Pall and Pall's customers would further EPT's and Dufresne's improper conduct as alleged herein and is therefore also barred by the defenses of illegality, unclean hands, and patent misuse.

67. Pall requests a judicial determination pursuant to the Federal Declaratory Judgments Act, Title 28, United States Code, Sections 2201 and 2202, of the respective rights of Pall and EPT and Dufresne with respect to EPT and Dufresne's claim that Pall and Pall's customers' use or sale of certain phosphate ester fluid ion exchange conditioning systems is an infringement of the '117 patent. Pall specifically requests a declaration that the '117 patent is invalid against Pall and Pall's customers and that neither the manufacture, use, importation, offer for sale, or sale of Pall's Ion Exchange Systems infringes any properly interpreted or valid claim of the '117 patent.

COUNT II - PATENT MISUSE

68. Pall realleges and incorporates herein by this reference paragraphs 1-67 of this Complaint, as though fully set forth herein.

69. Defendants have engaged in patent misuse by the acts alleged above and by, inter alia, improperly attempting to extend the limited exclusivity of the '117 patent beyond its scope, by improperly asserting that the use of Pall's Ion Exchange Systems infringes the '117 patent while knowing the '117 patent is invalid, unenforceable, and not infringed and lacking a reasonable basis to assert infringement, by asserting a patent obtained through fraudulent and/or inequitable conduct, and in reckless disregard of the facts and the harm that would be caused to Pall.

70. As a result of Defendants' unlawful and wrongful patent misuse, Pall is entitled to injunctive relief.

COUNT III - TRADE DISPARAGEMENT

71. Pall realleges and incorporates herein by this reference paragraphs 1-70 of this Complaint, as though fully set forth herein.

72. Pall has established a long-standing reputation for quality products based on its phosphate ester fluid ion exchange conditioning systems.

73. On information and belief, EPT and Dufresne in bad faith made knowingly false and misleading statements to Pall's customers and potential customers regarding Pall's Ion Exchange Systems, including the knowingly false statement that such products infringe the '117 patent, which Defendants know or should know is invalid, unenforceable, and not infringed.

74. EPT and Dufresne have disparaged the goods and business of Pall by false and misleading representations in violation of, inter alia, the Illinois Uniform Deceptive Trade Practice Act, 815 ILCS Section 510/2, similar statutes of other states in the U.S., and common law.

75. These representations are false and misleading because, as set forth above, Defendants and each of them knew or should have known that the '117 patent is invalid, unenforceable, and not infringed by Pall's Ion Exchange Systems.

76. These unfair deceptive trade practices have injured and will continue to injure the reputation of Pall and Pall's Ion Exchange Systems unless Defendants are enjoined from their unlawful conduct.

COUNT IV - LANHAM ACT SECTION 43(a)

77. Pall incorporates by reference and realleges the allegations set forth in paragraphs 1-76 of this Complaint, as though fully set forth herein .

78. EPT and Dufresne have made false and misleading statements of fact in various letters and other communications concerning the applicability, scope and validity of the '117 patent, EPT's products and Pall's products. These statements include: (a) false

statements about the applicability, scope and validity of the '117 patent that create a false impression that Defendants are the sole legitimate source of non-infringing phosphate ester fluid ion exchange conditioning systems; and (b) false statements that indicate that Pall's Ion Exchange Systems infringe the '117 patent.

79. These statements have deceived or are likely to deceive a substantial segment of the intended audience including Pall's actual and potential customers.

80. The deception is material and is likely to influence the purchasing decisions of the intended audience including Pall's actual and potential customers.

81. Pall's phosphate ester fluid ion exchange conditioning systems travel in, affect and involve interstate commerce in the United States.

82. As a direct and proximate result of EPT's and Dufresne's false and misleading statements, Pall has been and will continue to be injured in its business and property by such factors as, without limitation, business disruption, loss of present and future customers, loss of goodwill, loss of future sales to present and potential customers and other competitive disadvantages, which injuries are in an amount that has yet to be determined but will be established at trial.

83. Pall will continue to sustain injury and damage as a result of the Defendants' unlawful acts unless it is enjoined from continuing such unlawful conduct.

COUNT V - TORTIOUS INTERFERENCE WITH CONTRACTUAL RELATIONS AND PROSPECTIVE ECONOMIC ADVANTAGE

84. Pall realleges and incorporates herein by this reference paragraphs 1-83 of this Complaint, as though fully set forth herein.

85. Pall has a reputation for quality and valuable good will associated with its phosphate ester fluid ion exchange conditioning systems and has contractual and economic relationships with present and potential Pall customers, some of a longstanding nature. There is a reasonable likelihood that these customers, distributors and other prospective and potential customers and distributors will purchase phosphate ester fluid ion exchange conditioning systems from Pall in the future, with the probability of future economic benefit to Pall.

86. Defendants knew or should have known of the existence of Pall's contractual and prospective economic relationships and prospective economic relationships for the use, sale and purchase of Pall's Ion Exchange Systems.

87. On information and belief, EPT and Dufresne, willfully or recklessly sent or caused to be sent misleading letters concerning infringement of the '117 patent with the purpose and intent of disrupting the above described contractual and prospective economic relationships between Pall and Pall's customers and potential customers.

88. On information and belief, EPT and Dufresne, in bad faith, knew or should have known that the statements were false or misleading and that such statements would interfere with and disrupt the above-described contractual and prospective economic relationships between Pall and present and potential Pall customers.

89. As a direct and proximate result of the interference with and disruption of Pall's economic relationship by Defendants, some present Pall customers may cease or reduce their purchase of Pall's phosphate ester fluid ion exchange conditioning systems or demand compensation or indemnity. Prospective customers, similarly, may refrain from

purchase of Pall's phosphate ester fluid ion exchange conditioning systems because of the interference and disruption caused by Defendants' wrongful acts.

90. EPT's and Dufresne's misconduct did cause, is causing, and will continue to cause interference and disruption and damage to Pall's business.

COUNT VI - UNFAIR COMPETITION

91. Pall realleges and incorporates herein by this reference paragraphs 1-90 of this Complaint, as though fully set forth herein.

92. The above described acts and practices of Defendants constitute unfair methods of competition and unfair and deceptive acts and practices in violation of the Illinois Consumer Fraud and Deceptive Business Practices Act, 815 ILCS Section 505/2 and constitute wrongful acts and unfair competition under the statutes and common law of numerous other states.

93. As a result of Defendants' acts of unfair competition, Pall has been damaged in an amount not yet determined but will be established at trial.

94. Pall will continue to sustain injury and damages as a result of this unfair competition unless Defendants are enjoined from continuing their unlawful conduct.

95. In accordance with 815 ILCS Section 505/10a and similar laws in other states, Pall is entitled to recover damages and all such other relief that this Court deems proper.

PRAYER FOR RELIEF

WHEREFORE, Plaintiff Pall Corporation requests this Court to enter judgment against EPT and Dufresne as follows:

1. Declare that the '117 patent and each of its claims are invalid, unenforceable and not infringed by Pall or any Pall customers;

2. Declare this case to be "exceptional" within the meaning of 35 U.S.C. Section 285;

3. Award damages according to proof for the wrongs alleged herein;

4. Enter preliminary and permanent injunctive relief restraining EPT's and Dufresne's unlawful conduct and acts;

5. Award Pall its costs and reasonable attorneys' fees incurred in connection with this action;

6. Award such other relief as the Court may deem equitable and just.

JURY DEMAND

Pursuant to Federal Rules of Civil Procedure 38, Pall demands a jury trial on all issues triable of right by jury raised in this Complaint.

Dated: August 19, 2002

LEYDIG, VOIT & MAYER, LTD.

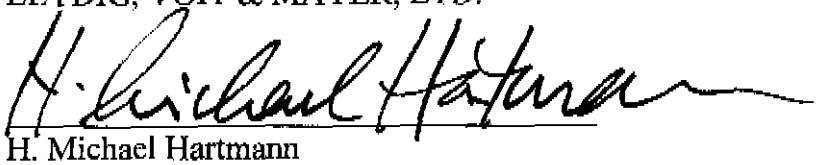
By: 

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Exhibit A

US005661117A

United States Patent [19]

Dufresne

[11] Patent Number: 5,661,117

[45] Date of Patent: Aug. 26, 1997

[54] **REGENERATION OF PHOSPHATE ESTER LUBRICATING FLUIDS**

[76] Inventor: **Peter Dufresne**, 428 Coachlight Bay S.W., Calgary, Alberta, Canada, T3H 1Z2

[21] Appl. No.: **421,771**

[22] Filed: **Apr. 14, 1995**

[51] **Int. Cl.**[6] **C10M 137/04**

[52] **U.S. Cl.** **508/433**; 58/150; 75/710

[58] **Field of Search** 252/49.8, 49.9; 508/433; 558/150; 75/710

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,708,508	1/1973	Schultz .	
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4,205,023	5/1980	Anzenberger	260/990
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4,302,335	11/1981	Habermas	210/651
4,741,857	5/1988	Horwitz et al. .	
5,364,452	11/1994	Cupertino et al.	423/22
5,464,551	11/1995	Deetman	252/78.5

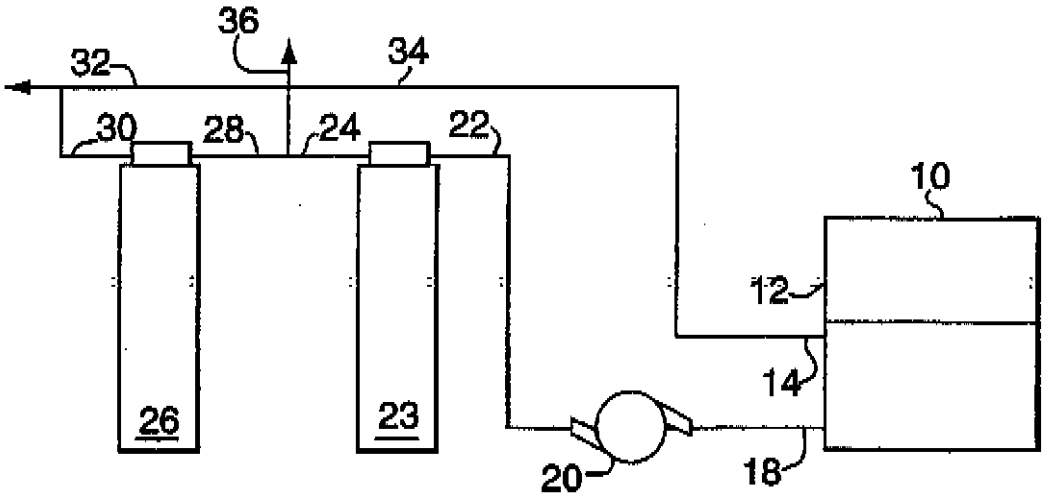
Primary Examiner—Jacqueline V. Howard

Attorney, Agent, or Firm—Paul Sharpe; McFadden, Fincham

[57] **ABSTRACT**

A method of removing contaminated phosphate ester materials is provided. The method involves the use of an anionic resin and a polymeric sorbent. The contaminated phosphate ester material is passed into contact with the anionic resin and optionally the sorbent. The method is particularly useful since it removes substantially all of the contaminants, generally metal material and acids, from the phosphate ester such that the phosphate ester can be reused for further applications.

16 Claims, 11 Drawing Sheets



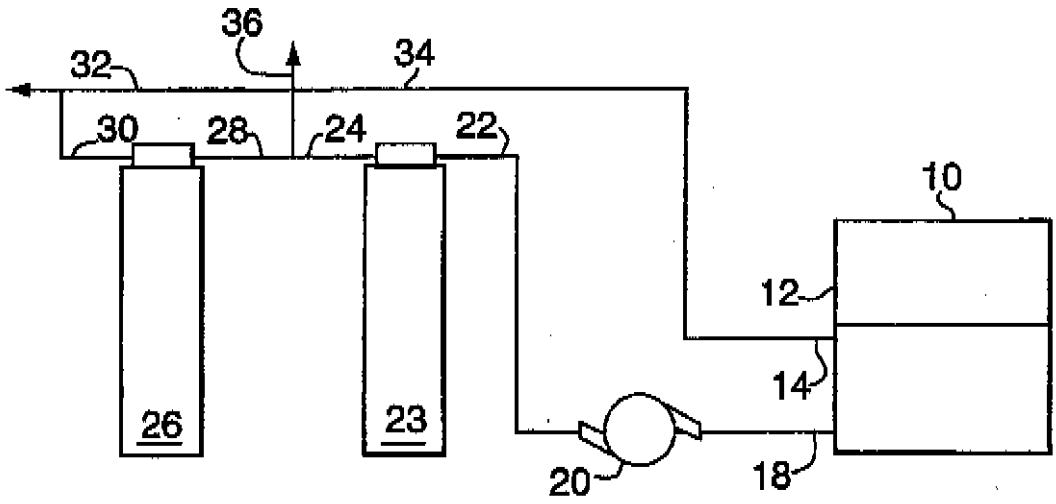


FIG. 1

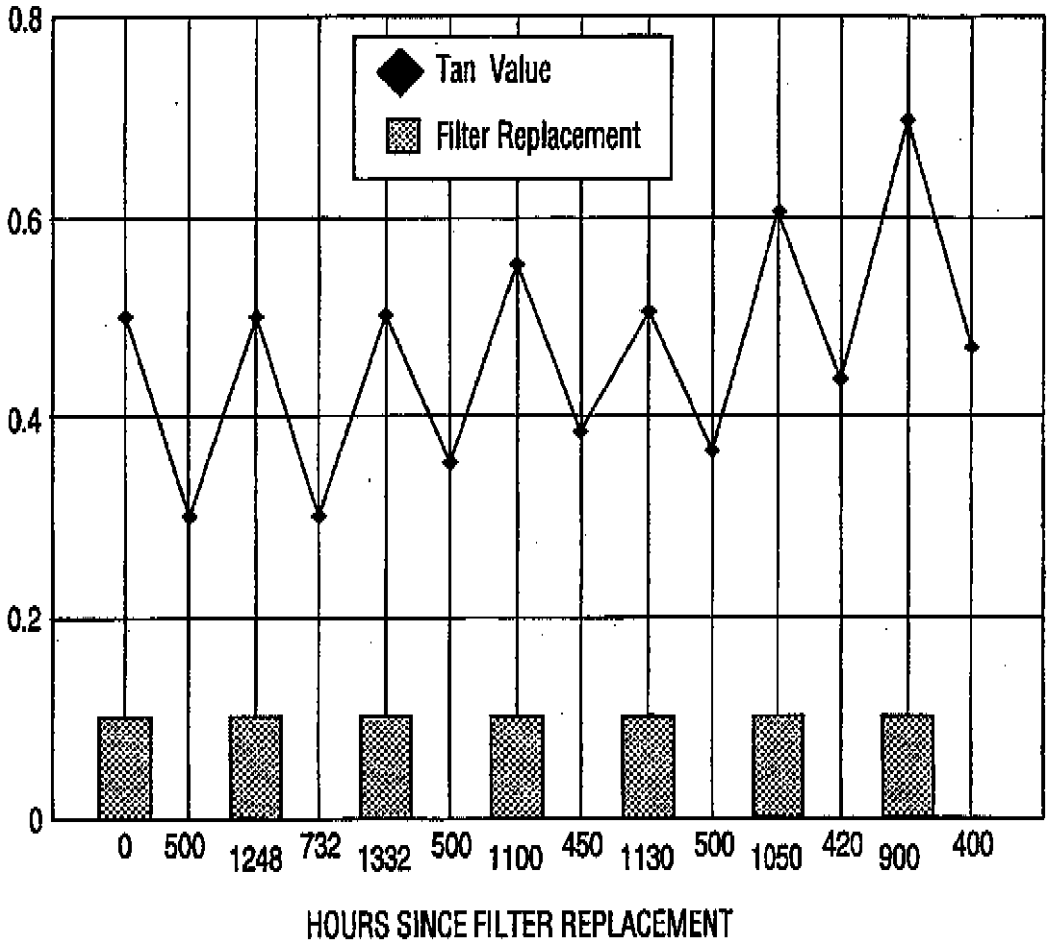

0.8
0.6
0.4
0.2
0
Tan Value
Filter Replacement
0
500
1248
732
1332
500
1100
450
1130
500
1050
420
900
400
HOURS SINCE FILTER REPLACEMENT

FIG. 2

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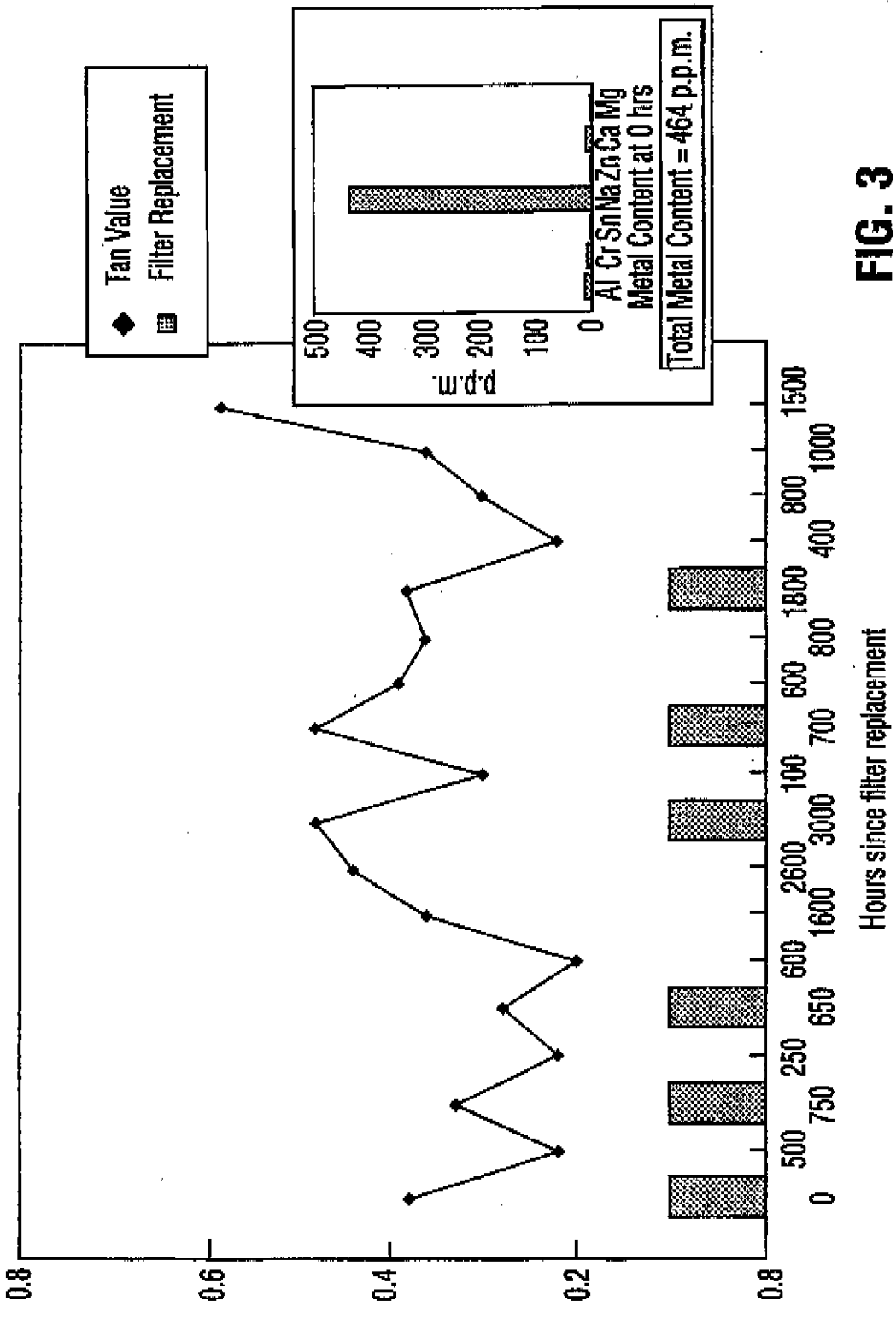

FIG. 3

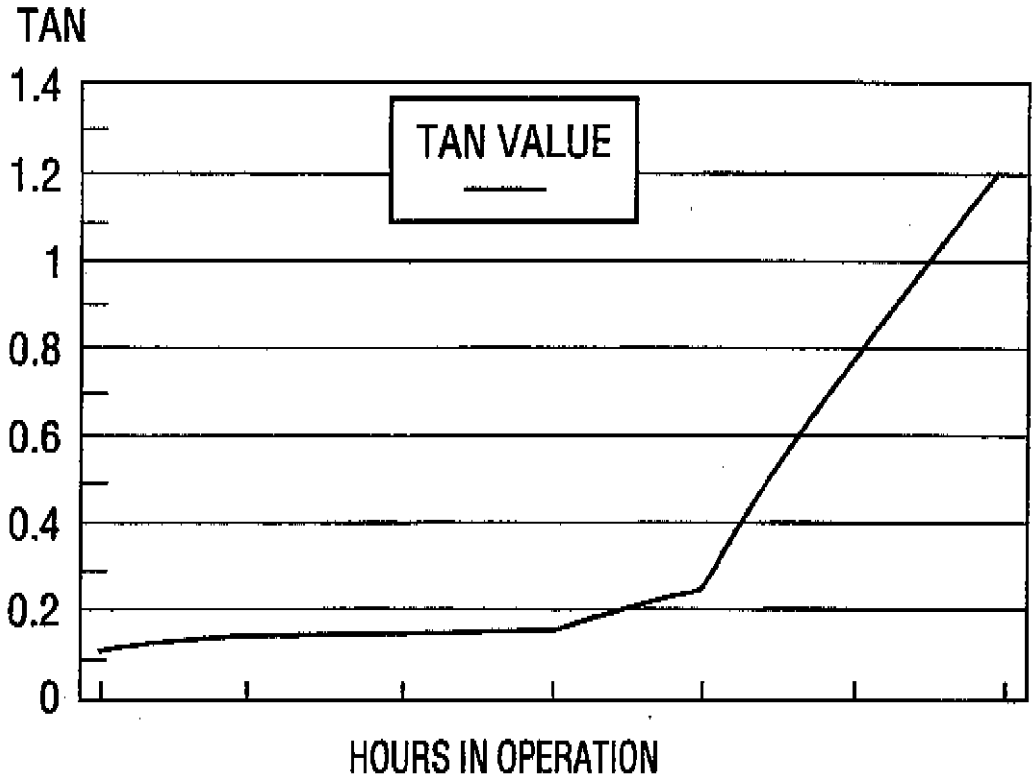
TAN
1.4
1.2
1
0.8
0.6
0.4
0.2
0
TAN VALUE
HOURS IN OPERATION

FIG.4

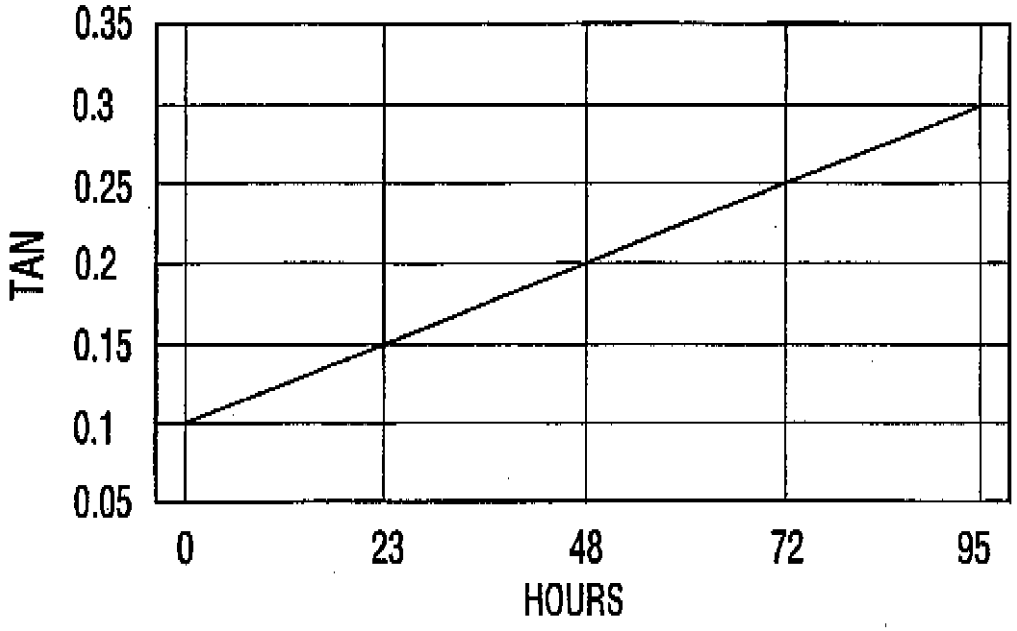
0.35
0.3
0.25
0.2
0.15
0.1
0.05
TAN
0
23
48
72
95
HOURS

FIG. 5

METAL	NEW FLUID	DEGRADED FLUID
	p.p.m.	p.p.m.
Al	0	13
Cr	7	9
Fe	0	1
Na	0	236
Si	6	0
Ca	1	4
Mg	1	1
Total	15	264

FIG. 6

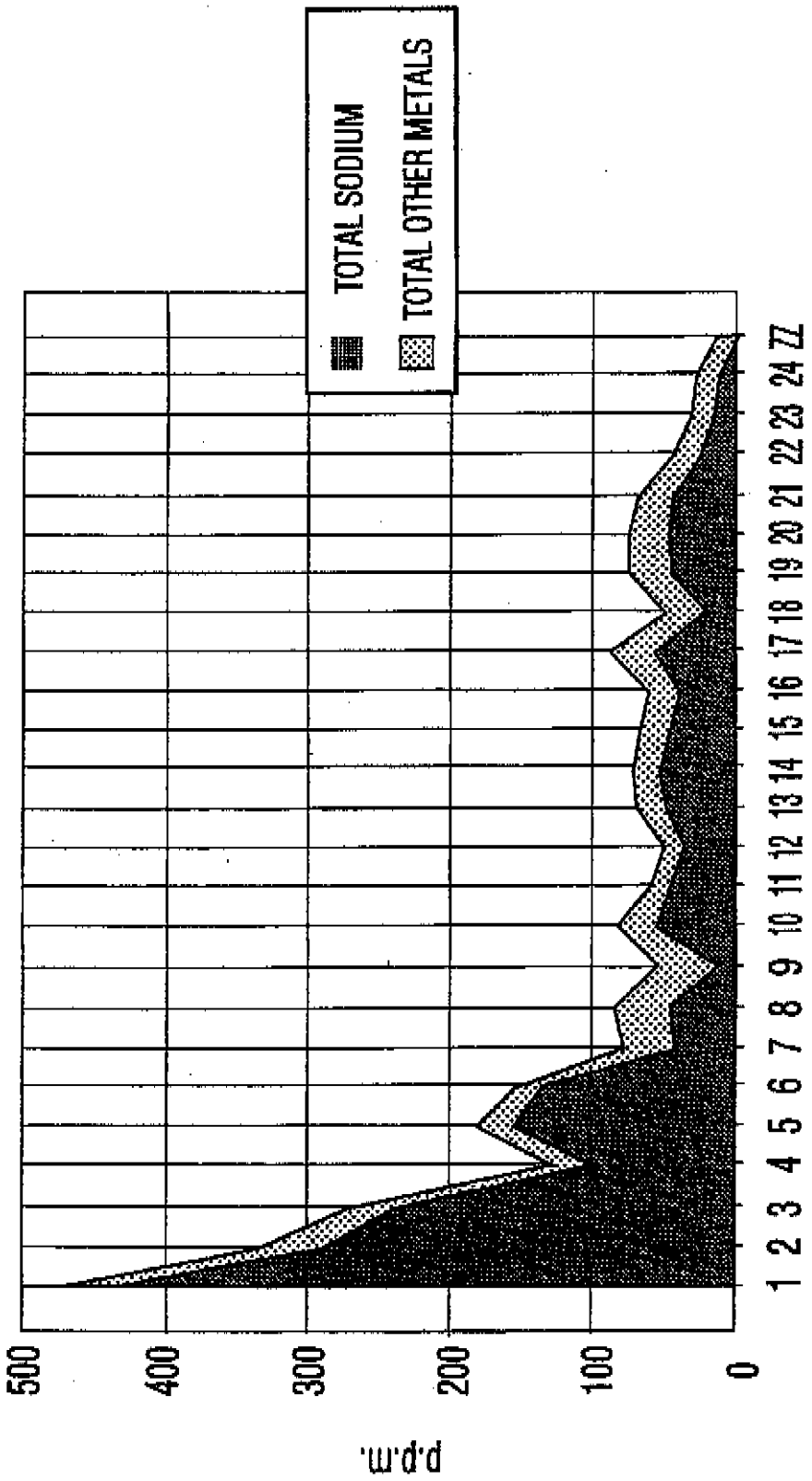
TOTAL SODIUM
TOTAL OTHER METALS
500
400
300
200
100
0
p.p.m.
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 ZZ
SAMPLE #

FIG. 7

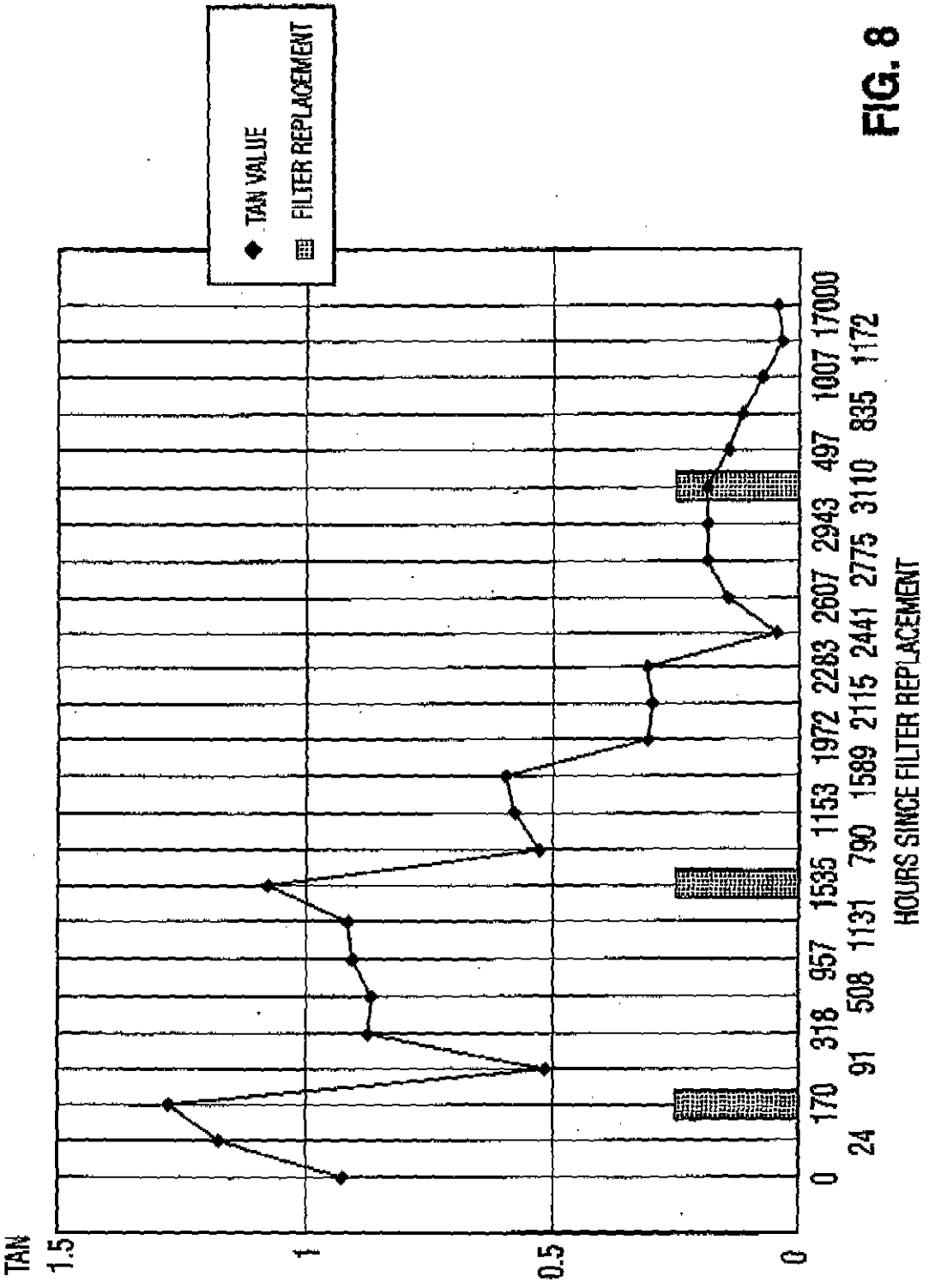
TAN VALUE
FILTER REPLACEMENT
TAN
1.5
1
0.5
0
0 24 91 170 318 508 957 1131 1535 790 1153 1589 1972 2115 2283 2441 2607 2775 2943 3110 497 835 1007 1172 17000
HOURS SINCE FILTER REPLACEMENT

FIG. 8

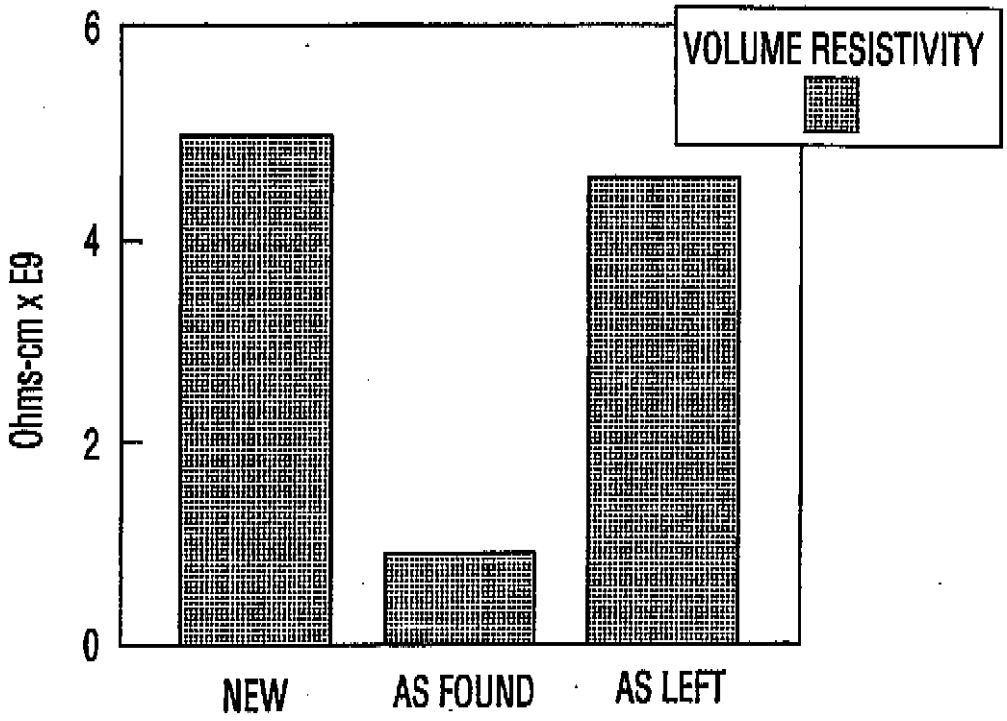
VOLUME RESISTIVITY
Ohms-cm x E9
6
4
2
0
NEW
AS FOUND
AS LEFT

FIG. 9

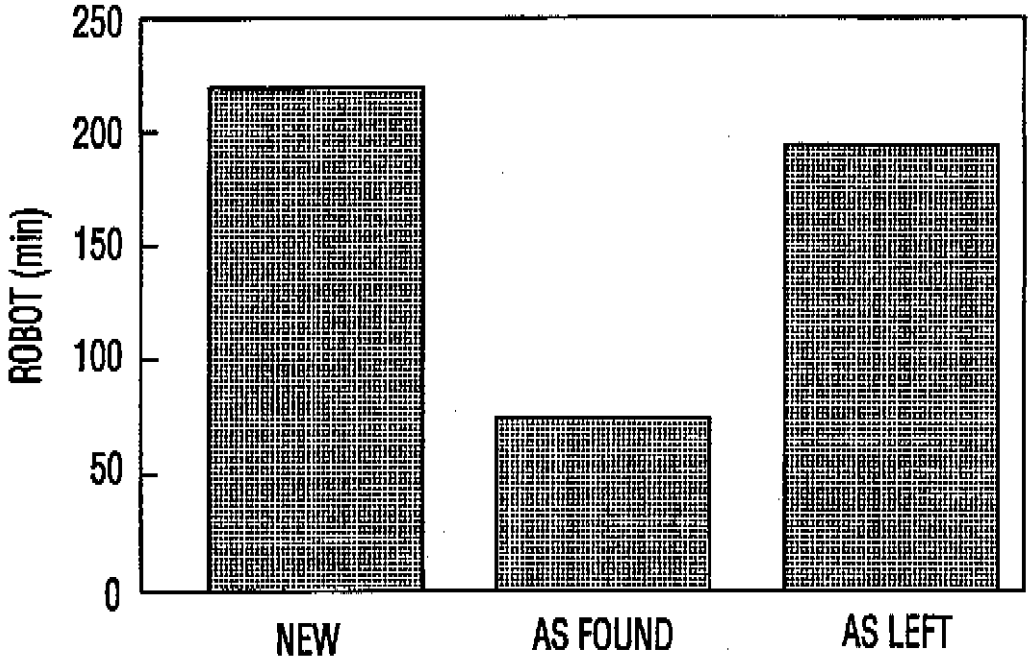
ROBOT (min)
250
200
150
100
50
0
NEW
AS FOUND
AS LEFT

FIG. 10

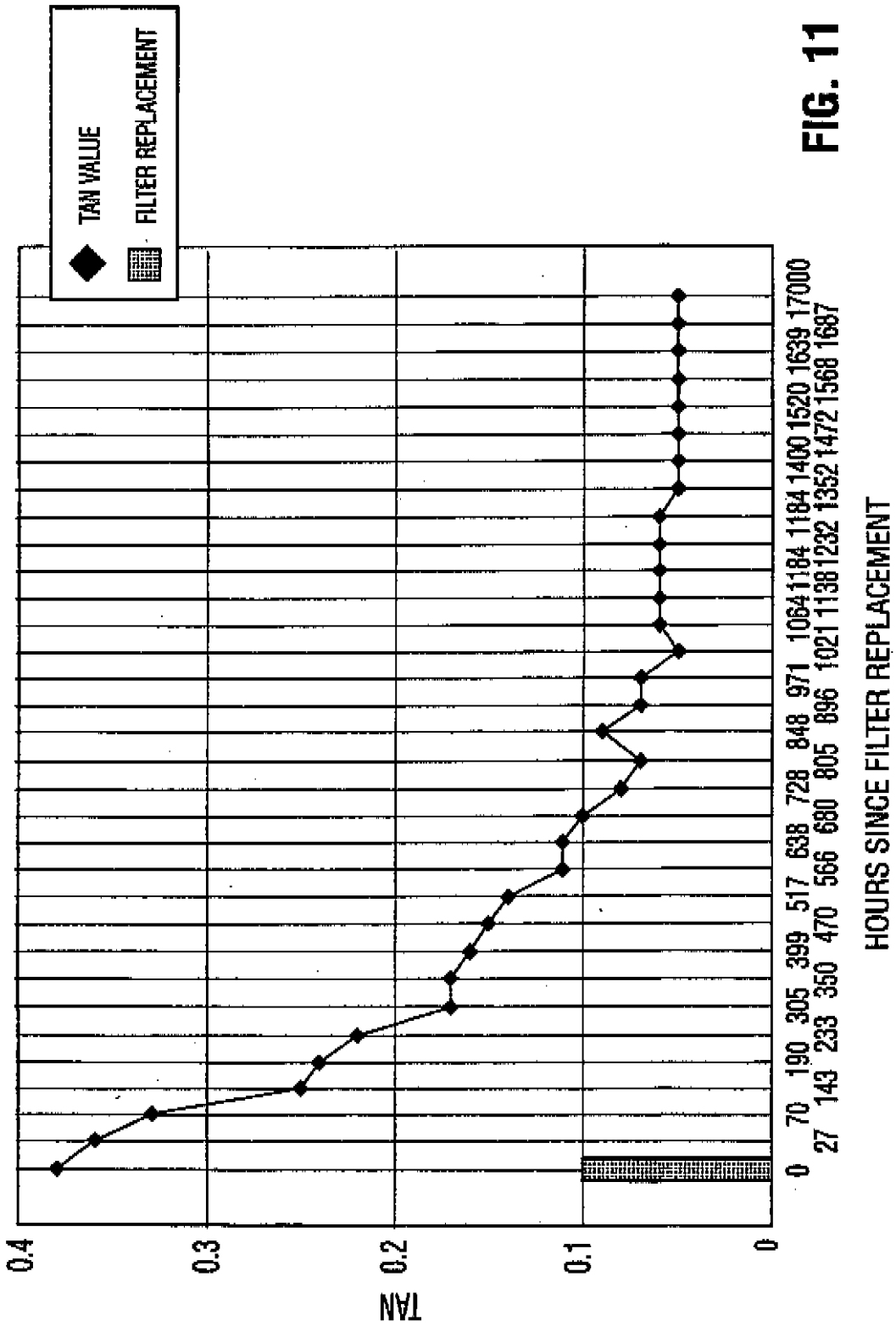
TAN VALUE
FILTER REPLACEMENT
TAN
0.4
0.3
0.2
0.1
0
0 27 70 143 190 233 305 350 399 470 517 566 638 680 728 805 848 896 971 1021 1064 1138 1184 1232 1352 1400 1472 1520 1568 1639 1687 17000
HOURS SINCE FILTER REPLACEMENT

FIG. 11

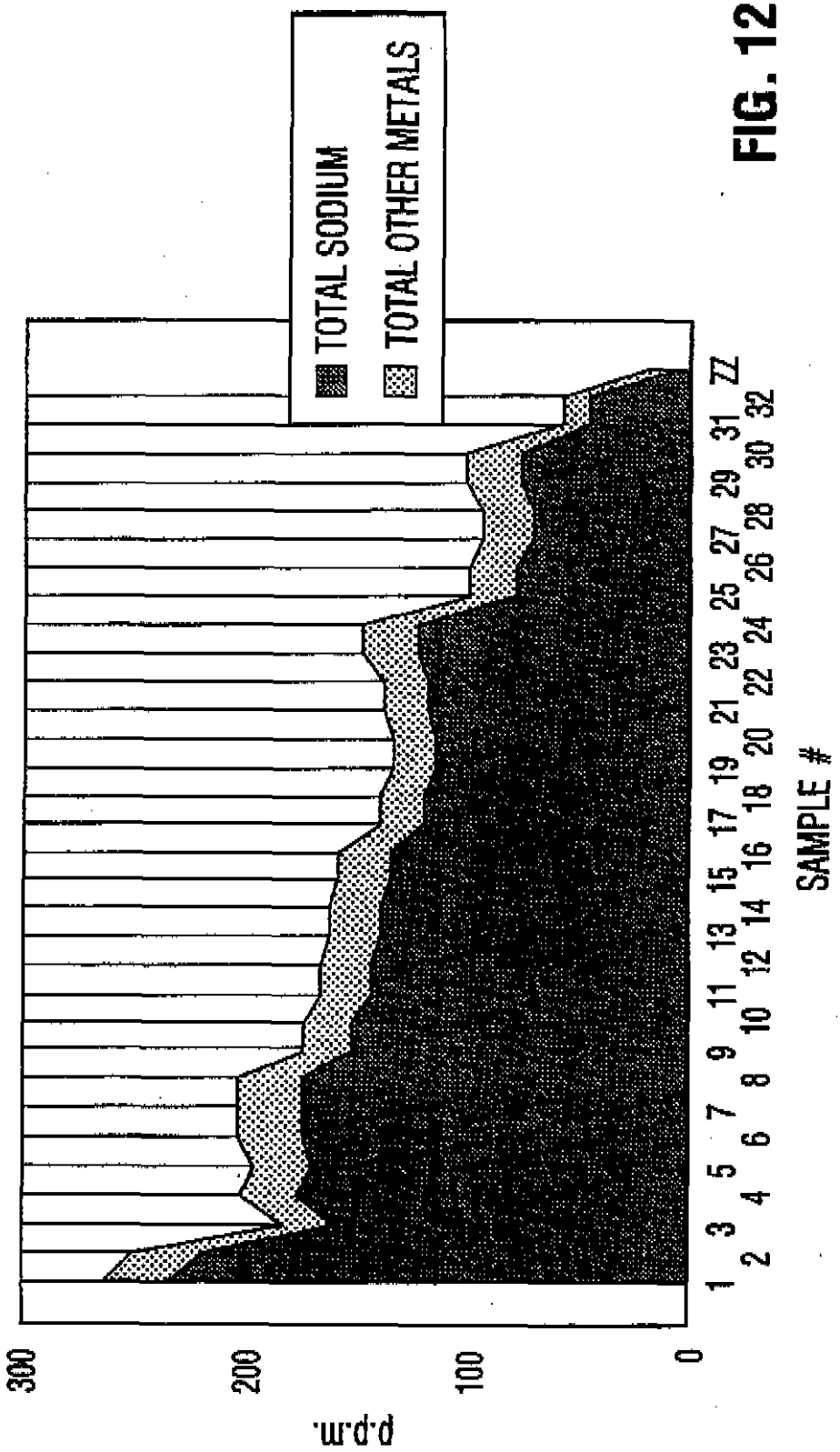
TOTAL SODIUM
TOTAL OTHER METALS
p.p.m.
300
200
100
0
SAMPLE #
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 ZZ

FIG. 12

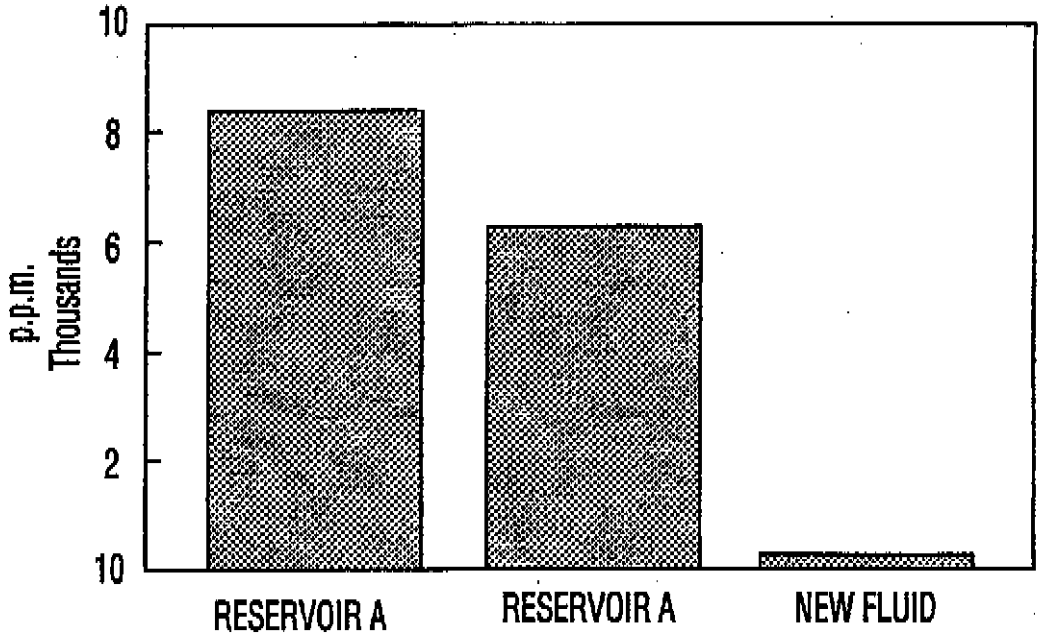

FIG. 13

REGENERATION OF PHOSPHATE ESTER LUBRICATING FLUIDS

FIELD OF THE INVENTION

The present invention relates to the regeneration of phosphate ester lubricating fluids and more particularly, the present invention is directed to the use of an anionic exchange resin for decontaminating such fluids.

BACKGROUND OF THE INVENTION

Generally speaking, gas turbine engines, steam turbines and other related hydraulic systems employ phosphate ester fluid lubricants, an example of which is phosphate ester fluid as the primary lubricating material. Although a particularly useful lubricant, the fluid is vulnerable to thermal degradation which results in the generation of acid contaminants in the form of phosphorus and phosphoric acids along with a variety of metal salts from acidic corrosion of internal gas turbine metals.

In an attempt to provide for possible cleaning methods, the prior art has provided filtration of the degraded fluid through Fuller's earth and/or activated alumina for the removal of acids from the thermal degradation. Recently, fluid filtration has progressed to continuous side stream treatment and has employed acid adsorbent medias which include activated alumina for acid removal.

Regarding the activated alumina and Fuller's earth, although these filter media are generally useful in the process of adsorbing acids, they contribute to the contamination level in the fluid and this has a significant impact on fluid quality and therefore operation of the apparatus employing this fluid. In the case of the Fuller's earth, adsorbed acids dissolve free calcium and magnesium which are naturally abundant in the Fuller's earth media. The calcium and magnesium enter the lubricating fluid as a soluble metal-salt and electrolytically plate out on hot engine components such as shafts, bearings and seals. The result is premature component wear and concomitant failure.

Similarly, activated alumina although generally understood to be a better adsorbent, additionally contributes sodium as a metal to the fluid. The problem is particularly pronounced when the sodium level becomes elevated beyond 90 parts per million (p.p.m.). At this level, or greater, the sodium has a tendency to react with the additional fluid degradation products in the lubricant to produce, for example, sodium phosphate and phosphites. Generally speaking, sodium phosphates chemically are commonly known as detergent soaps. The result can produce severe fluid foaming which, in turn, can cause lube oil pump cavitation as well as bearing and seal failures.

In an attempt to satiate the difficulties associated with decontaminating lubricant fluid of this variety, the art has proposed numerous methods, typical of which is indicated in U.S. Pat. No. 4,741,857, issued May 3, 1988 to Horwitz et al. Horwitz et al. teaches a method of purifying neutral organophosphorus extractants which primarily involves the mixing together of CMPO, TBP and NPH. The compounds are mixed together to form an organic extractant that is adapted to pick up the radiolytic and hydrolytic degradation products. In view of the fact that the acids are in both forms, i.e., salt and acid, the method requires both cation and anionic exchanges. The disclosure indicates that the extractants are contacted for at least 30 minutes with agitation. Further, the process is a two step process where the material to be treated must be contacted with the cation exchange in a first step to form a first solution and then subsequently contacted with the anion resin to complete the acid removal.

The above process is clearly limited in that it involves extensive treatment time and cannot produce a substantially contaminant free fluid rapidly and in a single pass through a single ionic exchange material.

Further prior art related to purification of fluids using ionic resins includes U.S. Pat. No. 3,708,508, issued to Schulz. The method is directed to the purification and recovery of tri-n-butylphosphate used in reprocessing nuclear fuel.

In view of what has been previously proposed in the art, it would be desirable to have a more efficient process where a spent or degraded lubrication fluid could be cleansed while in use without removal. This clearly has advantages in terms of reducing the probability of damage from using contaminated lubricant, clear cost savings since the material can be reused, as well as reducing the volume of chemical compounds which have to be handled carefully from an environmental point of view.

SUMMARY OF THE INVENTION

One object of the present invention is to provide an improved process for decontaminating and rejuvenating a lubricating fluid containing metal compounds and acidified contaminants.

A further object of the present invention is to provide a method of cleansing a phosphate ester lubricant contaminated with metal material and acids, the method comprising the steps of: providing a source of phosphate ester lubricant; providing a source of an anionic resin; passing the phosphate ester lubricant into contact with the resin; and removing the metal compounds and the acids with the resin to provide a substantially contaminant free reusable lubricating fluid.

Applicability of the method is widespread. The method can be employed to decontaminate and rejuvenate spent lubricating fluid or employed to rejuvenate newly manufactured phosphate fluid lubricants that do not meet new fluid specifications due to high acid levels or other such contamination. With respect to the latter point, this is particularly advantageous since in the prior art, the "off-spec" fluid could not be decontaminated using existing technology without recontaminating the fluid with additional metals, for example, magnesium and calcium, in the case of Fuller's earth.

Generally speaking, the contaminants typically found in lubricating fluids used in turbine engines include phosphoric and phosphorous acids along with various metal salt compounds formed from acidic corrosion of different metals utilized in gas engine turbine technology.

It has been found that use of an anionic resin is particularly useful for removing not only the metal salt compounds, but also for deacidifying the lubricating fluid. With passage of the contaminated fluid through the resin, new quality lubricating fluid has been created. As an optional processing step, the method may include a subsequent treatment of the decontaminated fluid with a polymeric ionic exchange sorbent. This is useful for removing any free phenols in the decontaminated lubricating fluid.

In one form, the anionic resin may comprise a polystyrene anionic resin, an example of which is Dowex M43 manufactured by the Dow Chemical Company.

The method may be practised in a continuous manner and may be employed with a turbine engine or hydraulic system where apparatus is attached directly to a suitable area of the machine. This permits the fluid to be continuously treated and therefore reduces the likelihood that the apparatus becomes damaged due to the use of a contaminated lubricating fluid.

Further, the fluid may be recirculated for several treatments or continuously. A further object of the present invention is to provide a system for decontaminating a bearing lubricant fluid contaminated with metal material and acids, the system comprising: at least one container for retaining an anionic resin, the container having an inlet for receiving a contaminated fluid therein and an outlet for discharging substantially contaminant free fluid; means for introducing the fluid into the inlet of the container; and means for recirculating the fluid from the outlet of the container into the inlet of the container for subsequent passage.

Having thus generally described the invention, reference will now be made to the accompanying drawings, illustrating preferred embodiments.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic illustration of the apparatus according to one embodiment of the present invention;

FIG. 2 is a graphical depiction of total acid number value versus filter replacement for interrupted treatment using activated alumina on reservoir A;

FIG. 3 is a graphical illustration of total acid number as a function of filter replacement for continuous treatment using activated alumina on reservoir A;

FIG. 4 is a graphical representation of the total acid number as a function of time illustrating the rate of change in total acid number (TAN) due to the hydrolytic stability of phosphate ester fluids;

FIG. 5 is a graphical representation of the oxidative stability as a function of total acid number which shows the rate of change in total acid number due to the oxidative stability of phosphate ester fluids;

FIG. 6 is a graphical representation of the total metals reduction in parts per million for various sample numbers taken from reservoir A;

FIG. 7 is a graphical representation of total metals reduction as a function of sample number;

FIG. 8 is a graphical representation of total acid number value as a function of filter replacement for reservoir A under continuous treatment using the Dowex M43 anion resin;

FIG. 9 is a graphical representation of the volume resistivity for fluids from reservoir A illustrating the change in fluid resistivity over the duration of the test and comparing with new fluid values;

FIG. 10 is a graphical representation of data generated from a rotary bomb oxidation test (RBOT) of the fluid from reservoir A before and after the resin test as compared with new fluid value;

FIG. 11 is a graphical representation of total acid number value as a function of replacement for the fluid from reservoir B under continuous filtration using Dowex M43 anion resin;

FIG. 12 is a graphical representation of total metal reduction expressed in parts per million as a function of the sample number for reservoir B; and

FIG. 13 is a histogram presentation of the phenol content for the fluid in reservoir A, reservoir B and that of a new fluid.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

FIG. 1 illustrates schematically one possible embodiment of the apparatus, globally denoted by numeral **10**. The lubricating fluid may be passed into a preliminary storage vessel **12** by inlet **14** thereon, the fluid being indicated in the reservoir **12** at **16**. It will be clearly understood that the lubricating fluid, may be fed into vessel **12** or further that vessel **12** may be directly connected to an apparatus employing the lubricating fluid which would permit real time treatment of the fluid.

Tubing, globally denoted by numeral **18**, permits fluid communication between the vessel **12** or other source of the fluid with the additional elements in the system to be discussed hereinafter. Fluid **16** is pumped through the system **10** via pump **20**, which pump **20** introduces the fluid to be treated into a first source of anionic resin at inlet **22**. Once the fluid has been passed through the source of anionic resin, the same is passed out outlet **24** of the anionic resin. The source of resin may comprise a column **23** or a resin bed or other suitable form of resin container.

Once treated with the anionic resin, the fluid may be directed to any number of possible routes. As a first possibility, the fluid may be then subsequently passed on to a sorbent treatment for removal of any remaining phenols and other residual contaminants. Numeral **26** denotes the source of polymeric sorbent which may be positioned in a conventional ionic exchange column and passed therein by inlet **28** from outlet **24** of anionic treatment area. Once the fluid has been circulated through the polymeric sorbent, it is passed out outlet **30** of column **26** and may subsequently be passed to an device (not shown) employing the fluid via line **32** or back to the reservoir **12** by line **34** for recirculation through the system **10**.

As a further possible alternative, once the fluid has been treated with the anionic exchange resin only and is passed out outlet **24**, the fluid may be then directed, via line **36**, to a device (not shown) which uses the fluid or recirculated through the system **10** via line **34**.

Any number of sources of resin **23** and sorbent **26** may be employed with the system. Further, these may be linked in series or parallel or any combination of these.

Turning to greater details of the present invention, the anionic resin, may comprise a polystyrene anionic resin and as an example, Dowex M43 as manufactured by the Dow Chemical Company may be a suitable solution for the resin. Others will be appreciated by those skilled in the art. Generally speaking, the Dowex M43 polystyrene resin has a free moisture content of approximately 50% to 55% and when used as part of a gas turbine fluid reclamation process, does not require drying prior to use. Oil flow is established as one imperial gallon per cubic foot of resin on a side stream basis. Phenols associated with the degraded phosphate ester fluid are removed with the use of the polymeric ion exchange sorbent as set forth in FIG. 1 and denoted by numeral **26**. This ion exchange sorbent has a very high internal surface area of up to 1000 square metres per gram (m^2g^{-1}). A preferred sorbent is Purolite MN-150.

In the lubricant field, lubricants for electrohydraulic control systems (EHC) are concerned with maintaining total acid number control (hereinafter referred to as TAN) for fluid lubricants. Generally, phosphate esters, isopropylphenyl phosphate ester (IPPP), and tertiary-butylphenyl phosphate (TBPP) are regarded as the choice compounds for lubrication purposes with phosphate-ester being an example. In the prior art, the previous methods of using Fuller's earth and alumina as well as other compounds such as diatomaceous earth etc. resulted in these processes being ineffectual to lower TAN levels to new fluid value, which must subscribe to a TAN value of 0.03.

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Having thus generally described the overall apparatus, reference will now be made to the accompanying example.

Two severely degraded phosphate ester reservoirs (A and B) were installed with M-43 anionic filters.

EXAMPLE

M-43 Anionic Exchange Resin Test on Reservoirs A & B

Both of the reservoirs each contained 1.0 cubic feet of resin and a fluid flow rate of 6 imperial gallons per minute was established therethrough. Oil samples for TAN and metals were taken frequently. Each of the reservoirs were fitted with filters bearing the M43 resin. The fluid that was employed for the purpose of this example was TBPP. The resin in each case was packaged into a standard Hillco filter housing which is normally used to hold 6 Fuller's earth, activated alumina or Selexsorb-GT cartridges that are 11" in diameter by 19" long. In the present invention, a new cartridge was designed maintaining the industrial standard dimension of 11"×19" to satisfy the requirements of optimising columnar height when using a resin.

The 11"×19" filter used in each of the reservoirs comprises the following components: a filter body manufactured from 20 gauge mild steel unperforated, a lid composed of 20 gauge mild steel perforated with a 100 mesh stainless screen spot welded on the inside of the lid. This was found effective to prevent resin beads from exiting the filter. An unperforated bottom on the filter body with a centered drain tube attached was employed. The length of the tube was 2".

The filters as manufactured in this process have virtually no shelf life. due to the high water content of the M-43 resin and they are manufactured on an as required basis for immediate installation. Filter life is dependant on the severity of the turbine application, but typically the life varies from a minimum of 16 months to a maximum of 27 months. The filters are changed when oil lab analysis shows an increase in TAN above 0.07.

The design of the filter herein allows oil flow to enter the filter body through the holes in the top lid where it flows down through the resin at about 20 p.s.i.g. and exits the filter body through the holes in the bottom of the filter center tube.

For illustrative purposes, FIG. 2 illustrates TAN value as a function of filter replacement on reservoir A using interrupted filtration and activated alumina. As is evident from the data, it can be noted that after approximately 3000 hours of fluid life, a TAN value of 0.30 cannot be maintained and the TAN value gradually increases. In spite of numerous replacements of the activated alumina cartridges, the data clearly illustrates that the TAN value of the fluid continued to increase over the life of the fluid.

Under similar conditions as in FIG. 2. FIG. 3 illustrates data with respect to the activated alumina in reservoir A, but for continuous filtration. The overall fluid life slightly improved, however, after approximately 8500 hours of operation, the fluid TAN reached 0.58. At this point in time, 8 sets of cartridges had been exhausted in an attempt to minimize TAN and cartridge maintenance costs approached 60% of the value of the fluid in the first year of operation. Total fluid metals as illustrated in the inset exceeded 400 p.p.m. Further, acid formation escalated at such a rate that the activated alumina could not reduce overall acid levels.

FIGS. 4 and 5 illustrate data directed to the hydrolytic stability the function of total acid number and the oxidative stability as a function of total acid number. In each case, the rate of change in the TAN is shown for phosphate ester fluids.

FIG. 6 illustrates the metal analysis of new and degraded TBPP fluid. Phosphorus is not included due to the nature of the fluid. All analysis shows phosphorus at over 10,000 p.p.m.

Metal analysis indicated that calcium, magnesium, aluminum, iron, sodium and silicon are present in degraded fluid. Calcium and magnesium resulted from some use over time of the Fuller's earth adsorbent. Aluminum resulted from improper installation of a half micron filter downstream of the activated alumina sorbent, thereby allowing the alumina media to migrate into the lube oil system. The presence of the iron is the result of a small amount of oxidation of the mild steel filters that resulted from a five day delay in installation of the filters after manufacture. The presence of the sodium is the result of numerous activated alumina filter cartridge change outs over a three year period. Introduction of free sodium into the oil system is proportional to the number of activated alumina filter change outs, and exponential to the TAN level of the fluid. Increase in sodium tends to follow the graph of the oxidative stability of the fluid as set forth herein previously with respect to FIG. 5. The presence of silicon is due to the addition thereof as an anti-foam agent. This can vary from 1 to 6 p.p.m.

Turning to FIG. 7, shown is a graphical representation of total metals reduction as a function of sample number taken over a period of time. Data is illustrated for a total sodium and total other metals.

The test on reservoir A was complicated due to the fact that the first set of ion exchange filters that were installed were filled with Dowex M21, a cationic resin. The graph reveals that at 170 hours into the test, the TAN had increased to 1.28 with metals being lowered significantly to 268 p.p.m. New anionic M-43 filters were installed and the TAN was lowered to 0.52 before exhaustion of the filters. The filters were not changed until 1535 hours into the test. The filters were changed with M-43 resin. Both metals and TAN value decreased until 2441 hours into the test at which time the filters were exhausted. They were changed at 3110 hours and metals and TAN value were lowered to near new fluid values. FIG. 8 illustrates the TAN value as a functional filter replacement for the hours set forth herein above.

As a further illustration of the utility of the present invention, FIG. 9 illustrates the volume resistivity for a new fluid, a fluid prior to treatment with the anionic resin and for the fluid subsequent to treatment. As is clearly evident in the histogram, the fluid as treated is extremely close to new fluid resistivity values. Similarly, FIG. 10 sets forth a similar comparison on a rotary bomb oxidation test, which test is indicative as to how oxidized fluid is. Clearly, subsequent to treatment with the anionic resin, the treated fluid substantially approximates the oxidation level of the new fluid.

Table 1 illustrates detailed lab analysis data for various test runs for reservoir A using the Dowex M43 resin. Data is tabulated for TAN value, water content, total metal content and a breakdown of individual metals, namely aluminum, chromium, tin, iron, sodium, calcium, magnesium, zinc and silicon.

Turning to graphical data for reservoir B, FIG. 11 graphically illustrates the TAN value as a function of filter replacement for reservoir B under continuous filtration using the Dowex M43 anion resin.

FIG. 12 graphically illustrates the total metals reduction for reservoir B under continuous filtration with data specifically being illustrated for total sodium content as well as a total for other metals in the fluid. There is a clear and steady decline of metal concentration in the fluid over the course of time with the data being exemplary at sample ZZ taken at 17000 hours.

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FIG. 13 is a histogram presentation of the phenol content in parts per million for reservoirs A and B as compared to new fluid. This data depicts the phenol content prior to treatment with the sorbent (to be discussed hereinafter).

Generally speaking, high phenol values are indicative of fluid deterioration. In the gas turbine application, the phenols do not need to be removed, but if these compounds are removed, the result is slight improvements to fluid resistivity values, colour and other fluid thermal degradation tests. FIG. 13 illustrates the rate of phenol removal in parts per million using the Purolite MN-150 sorbent.

Data similar to that set forth in Table I with respect to reservoir A is set forth in Table 2 for reservoir B.

Based on the tests conducted on the two degraded phosphate ester fluids used as a main bearing lubricant in gas turbine applications, the use of the polystyrene anionic resin can clearly be said to regenerate severely deteriorated reservoirs to at least 95% of new fluid quality. It is clear that this has significant advantages in terms of eliminating expensive fluid replacement and unnecessary removal of the fluid.

Although embodiments of the invention have been described above, it is not limited thereto and it will be apparent to those skilled in the art that numerous modifications form part of the present invention insofar as they do not depart from the spirit, nature and scope of the claimed and described invention.

TABLE 1

					Detailed Lab Analysis Reservoir A								
	Hrs run	Hrs recycled	Tan	Water	Total Metals	Aluminum	Cr	Sn	Iron	Sodium	Calcium	Magnesium	Zinc	Si
1	79919	0	0.93	481	475	10	6	3	0	435	8	1	12	0
2	79937	24	1.18	1216	326	8	6	2	3	290	6	0	11	0
3	80083	170	1.28	228	268	6	7	2	3	238	7	0	5	0
4	80174	91	0.52	3138	124	6	6	0	4	100	3	0	5	0
5	80401	318	0.88	102	181	3	8	0	3	160	5	0	2	0
6	80591	508	0.87	116	156	4	7	0	3	138	3	0	1	0
7	81040	957	0.91	3224	75	1	8	2	14	44	5	0	1	0
8	81214	1131	0.92	3720	85	3	10	0	18	47	3	2	2	0
9	81618	1535	1.08	3986	50	1	7	3	21	12	5	0	1	1
10	82408	790	0.53	468	83	1	9	0	10	57	4	0	2	0
11	82771	1153	0.58	531	59	1	7	0	4	45	2	0	0	1
12	83207	1589	0.6	324	50	1	6	1	4	36	2	0	0	0
13	83590	1972	0.31	222	70	5	7	2	3	51	2	0	0	1
14	83733	2115	0.3	361	72	5	6	1	3	55	2	0	0	1
15	83901	2283	0.31	336	68	8	7	0	2	48	2	1	0	0
16	84039	2441	0.05	185	60	8	7	0	2	40	2	1	0	0
17	84225	2607	0.15	260	87	13	10	0	4	57	3	0	0	1
18	84393	2775	0.19	308	45	12	8	0	4	20	0	1	0	0
19	84561	2943	0.19	291	75	12	9	0	3	48	2	1	0	0
20	84728	3110	0.2	319	76	12	7	0	4	50	2	1	0	0
21	85225	497	0.15	262	69	11	7	0	3	45	2	1	0	0
22	85563	835	0.12	696	44	7	6	0	3	27	1	0	0	0
23	85735	1007	0.08	210	33	6	6	0	2	18	1	0	0	0
24	85900	1172	0.04	136	30	6	6	0	2	15	1	0	0	0
ZZ	98067	17000	0.05	84	16	5	6	0	1	3	0	1	0	1

TABLE 2

					Detailed Lab Analysis Reservoir B							
	Hrs run	Hrs recycled	Tan	Water	Total Metals	Al	Cr	Sn	Fe	Na	Ca	Mg	Si
1	88487	0	0.38	86	264	13	9	0	1	236	4	1	0
2	88514	27	0.36	214	251	13	10	1	1	219	4	1	2
3	88557	70	0.33	132	182	10	7	1	1	159	3	0	1
4	88630	143	0.25	104	203	11	8	1	1	178	3	0	1
5	88677	190	0.24	118	196	11	8	1	1	171	3	0	1
6	88720	233	0.22	106	204	12	10	2	2	175	3	0	0
7	88792	305	0.17	154	204	12	10	2	2	175	3	0	0
8	88837	350	0.17	181	204	12	10	2	2	175	3	0	0
9	88886	399	0.16	173	175	10	8	0	1	153	3	0	0
10	88957	470	0.15	105	175	10	8	0	1	153	3	0	0
11	89004	517	0.14	83	168	10	8	0	2	145	3	0	0
12	89053	566	0.11	99	168	10	8	0	2	145	3	0	0
13	89125	638	0.11	88	163	10	8	0	3	140	1	1	0
14	89167	680	0.1	117	163	10	8	0	3	140	1	1	0
15	89215	728	0.08	122	160	10	8	0	2	136	3	1	0
16	89292	805	0.07	59	160	10	8	0	2	136	3	1	0
17	89335	848	0.09	80	141	7	7	0	1	121	3	2	0
18	89383	896	0.07	74	141	7	7	0	1	121	3	2	0

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TABLE 2-continued

				Detailed Lab Analysis Reservoir B									
	Hrs run	Hrs recycled	Tan	Water	Total Metals	Al	Cr	Sn	Fe	Na	Ca	Mg	Si
19	89458	971	0.07	65	136	7	7	0	1	116	3	2	0
20	89508	1021	0.03	96	136	7	7	0	1	116	3	2	0
21	89551	1064	0.06	121	140	7	7	0	1	120	3	2	0
22	89623	1138	0.06	107	140	7	7	0	1	120	3	2	0
23	89671	1184	0.06	81	150	10	7	2	2	124	4	1	0
24	89719	1232	0.06	89	150	10	7	2	2	124	4	1	0
25	89671	1184	0.06	139	102	8	5	1	2	80	5	1	0
26	89839	1352	0.05	152	102	8	5	1	2	80	5	1	0
27	89887	1400	0.05	126	96	8	5	4	2	72	4	1	0
28	89959	1472	0.05	174	96	8	5	4	2	72	4	1	0
29	90007	1520	0.05	242	103	8	6	1	2	78	4	4	0
30	90055	1568	0.05	218	103	8	6	1	2	78	4	4	0
31	90126	1639	0.05	198	60	6	6	0	0	47	1	0	0
32	90174	1687	0.05	205	60	6	6	0	0	47	1	0	0
22	107174	17000	0.05	120	17	5	6	0	1	3	0	1	1

I claim:

1. A method of cleansing a phosphate ester lubricant fluid contaminated with metal material and phosphorous based acids to a new fluid quality having a total acid number of 0.03, said method consisting essentially of:

providing a source of phosphate ester lubricant contaminated with at least one metal selected from the group consisting of aluminum, chromium, tin, iron, sodium, calcium, magnesium and silicon and phosphorous based acids;

providing a source of an anionic resin;

passing said phosphate ester lubricant fluid contaminated with said metal and said acids into contact with said resin; and

removing said metal compounds and said acids with said resin to provide a substantially contaminant free reusable lubricatant fluid of a new fluid quality having a total acid number of 0.03.

2. The method as set forth in claim 1, further including an additional filtration step of passing said substantially contaminant free lubricating fluid through a source of a polymeric sorbent for removal of any remaining contaminants.

3. The method as set forth in claim 1, further including the step of recirculating said substantially contaminant free lubricating fluid through said anionic resin.

4. The method as set forth in claim 2, further including the step of recirculating the fluid having been exposed to the said additional filtration step through said system.

5. The method as set forth in claim 1, wherein said method is a continuous method.

6. The method as set forth in claim 1, wherein said phosphate ester lubricating fluid comprises an isopropylphenyl phosphate ester.

7. The method as set forth in claim 1, wherein said phosphate ester lubricating fluid comprises tertiarybutylphenyl phosphate.

8. The method as set forth in claim 1, wherein said acids include phosphorous and phosphoric acid.

9. A method of regenerating a phosphate ester lubricatant fluid contaminated with metal material selected from the group consisting of aluminum, chromium, tin, iron, sodium, calcium, magnesium and silicon and phosphorous based acidified contaminants to new fluid quality having a total acid number of 0.03, consisting essentially of:

providing a source of a polystyrene anionic resin;

passing said contaminated fluid into contact with said polystyrene anionic resin to remove said metal material and said acidified contaminants to regenerate said lubricating fluid to a substantially contaminant free reusable lubricating fluid of a new fluid quality having a total acid number of 0.03.

10. The method as set forth in claim 9, further including the step of passing fluid treated with said anionic resin into contact with a polymeric sorbent resin.

11. The method as set forth in claim 9, wherein said polystyrene anionic resin comprises Dowex™ M43 anionic resin.

12. The method as set forth in claim 10, wherein said polymeric sorbent comprises Purolite MN-150 polymeric ion exchange sorbent.

13. The method as set forth in claim 9, wherein said phosphate ester lubricating fluid comprises isopropylphenyl phosphate ester.

14. The method as set forth claim 9, wherein said phosphate ester lubricating fluid comprises tertiarybutylphenol phosphate.

15. The method as set forth in claim 9, wherein said method is a continuous method.

16. The method as set forth in claim 9, wherein said method is a closed circuit method.

* * * * *

Exhibit B

US005661117B1

REEXAMINATION CERTIFICATE (4142nd)

United States Patent [19] [11] **B1 5,661,117**

Dufresne [45] Certificate Issued **Aug. 29, 2000**

[54] **REGENERATION OF PHOSPHATE ESTER LUBRICATING FLUIDS**

[76] Inventor: **Peter Dufresne**, 428 Coachlight Bay S.W., Calgary, Alberta, Canada, T3H 1Z2

Reexamination Request:
No. 90/005,088, Aug. 26, 1997

Reexamination Certificate for:

Patent No.:	5,661,117
Issued:	Aug. 26, 1997
Appl. No.:	08/421,771
Filed:	Apr. 14, 1995

[51] **Int. Cl.**[7] **C10M 137/04**
[52] **U.S. Cl.** **508/433**; 558/150; 75/710
[58] **Field of Search** 508/433; 558/150; 75/710

[56] **References Cited**

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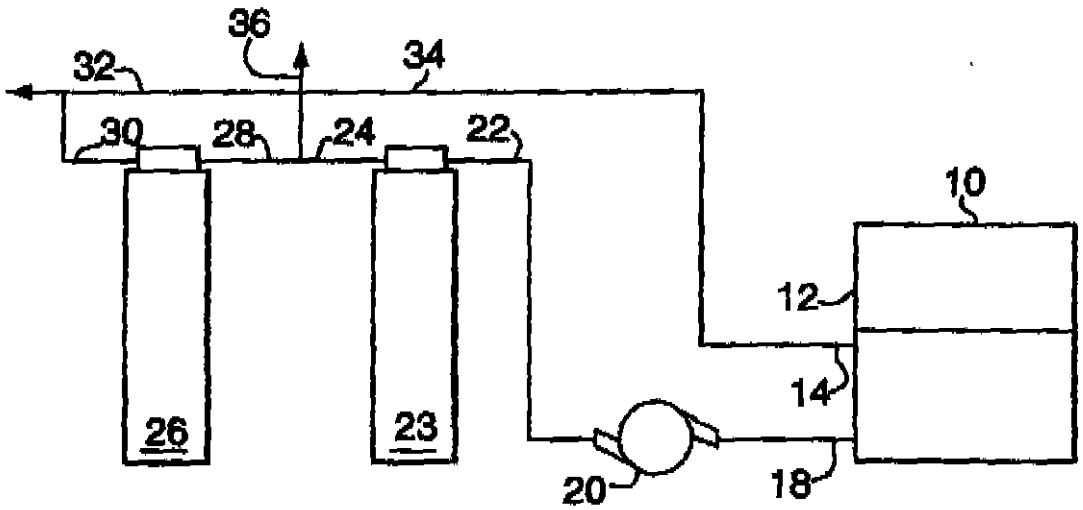
Primary Examiner—J V Howard

[57] **ABSTRACT**

A method of removing contaminated phosphate ester materials is provided. The method involves the use of an anionic resin and a polymeric sorbent. The contaminated phosphate ester material is passed into contact with the anionic resin and optionally the sorbent. The method is particularly useful since it removes substantially all of the contaminants, generally metal material and acids, from the phosphate ester such that the phosphate ester can be reused for further applications.



UNITED STATES DISTRICT COURT
NORTHERN DISTRICT OF ILLINOIS

Civil Cover Sheet

This automated JS-44 conforms generally to the manual JS-44 approved by the Judicial Conference of the United States in September 1974. The data is required for the use of the Clerk of Court for the purpose of initiating the civil docket sheet. The information contained herein neither replaces nor supplements the filing and service of pleadings or other papers as required by law. This form is authorized for use only in the Northern District of Illinois.

Plaintiff(s): PALL CORPORATION

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Plaintiff's Atty: H. Michael Hartmann
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312-616-5600

Defendant(s):ENVIRONMENTAL AND POWER TECHNOLOGIES, LTD. and PETER DUFRESNE

County of Residence: Alberta, Canada

Defendant's Atty:

DOCKETED
AUG 2 0 2002

02C 5898

II. Basis of Jurisdiction: **3. Federal Question (U.S. not a party)**

JUDGE GOTTSCHALL

III. Citizenship of Principal Parties **(Diversity Cases Only)**
Plaintiff:- **N/A**
Defendant:- **N/A**

MAGISTRATE JUDGE SCHENKIER

IV. Origin : **1. Original Proceeding**

V. Nature of Suit: **830 Patent**

VI.Cause of Action: **Declaratory Judgment Action pursuant to 28 U.S.C. Sections 2201-2202 and 35 U.S.C. Sections 1, et seq.**

VII. Requested in Complaint
Class Action:
Dollar Demand:
Jury Demand: **Yes**

VIII. This case **IS NOT** a refiling of a previously dismissed case.

Signature: H. Michael Hartmann

Date: 8/19/02

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UNITED STATES DISTRICT COURT
NORTHERN DISTRICT OF ILLINOIS
Eastern Division

In the Matter of

PALL CORPORATION
v.
ENVIRONMENTAL AND POWER
TECHNOLOGIES, LTD. and PETER DUFRESNE

Case Number: 02C 5898

DOCKETED
AUG 20 2002
JUDGE GOTTSCHALL

APPEARANCES ARE HEREBY FILED BY THE UNDERSIGNED AS ATTORNEY(S) FOR:

PALL CORPORATION

MAGISTRATE JUDGE SCHENKIER

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